FORM PTO-1390 (Modified) (REV 11-2000) U.S. DEPARTMENT OF COMMERCE PATENT AND TRADEMARK OFFICE

TRANSMITTAL LETTER TO THE UNITED STATES DESIGNATED/ELECTED OFFICE (DO/EO/US) CONCERNING A FILING UNDER 35 U.S.C. 371

ATTORNEY'S DOCKET NUMBER: 4004-030-30

U.S. APPLICATION NO (IF KNOWN, SEE 37 CFR): 10/069130

INTERNATIONAL APPLICATION NO.	INTERNATIONAL FILING DATE	PRIORITY DATE CLAIMED
PCT/EP00/08205	21 AUGUST 2000	26 AUGUST 1999

TITLE OF INVENTION: **GLAZING**

APPLICANT(S) FOR DO/EO/US: **DEGAND, Etienne**

Applicant herewith submits to the United States Designated/Elected Office (DO/EO/US) the following items and other information:

1. ☒ This is a **FIRST** submission of items concerning a filing under 35 U.S.C. 371.
2. ☐ This is a **SECOND** or **SUBSEQUENT** submission of items concerning a filing under 35 U.S.C. 371.
3. ☒ This is an express request to begin national examination procedures (35 U.S.C. 371(f)). The submission must include itens (5), (6), (9) and (24) indicated below.
4. ☒ The US has been elected by the expiration of 19 months from the priority date (Article 31).
5. ☒ A copy of the International Application as filed (35 U.S.C. 371 (c) (2))
   a. ☐ is attached hereto (required only if not communicated by the International Bureau).
   b. ☒ has been communicated by the International Bureau.
   c. ☐ is not required, as the application was filed in the United States Receiving Office (RO/US).
6. ☐ An English language translation of the International Application as filed (35 U.S.C. 371(c)(2)).
   a. ☐ is attached hereto.
   b. ☐ has been previously submitted under 35 U.S.C. 154(d)(4).
7. ☒ Amendments to the claims of the International Application under PCT Article 19 (35 U.S.C. 371 (c)(3))
   a. ☐ are attached hereto (required only if not communicated by the International Bureau).
   b. ☐ have been communicated by the International Bureau.
   c. ☐ have not been made; however, the time limit for making such amendments has NOT expired.
   d. ☒ have not been made and will not be made.
8. ☐ An English language translation of the amendments to the claims under PCT Article 19 (35 U.S.C. 371(c)(3)).
9. ☐ An oath or declaration of the inventor(s) (35 U.S.C. 371 (c)(4)).
10. ☐ An English language translation of the annexes to the International Preliminary Examination Report under PCT Article 36 (35 U.S.C. 371 (c)(5)).
11. ☒ A copy of the International Preliminary Examination Report (PCT/IPEA/409).
12. ☒ A copy of the International Search Report (PCT/ISA/210).

Items 13 to 20 below concern document(s) or information included:

13. ☐ An Information Disclosure Statement under 37 CFR 1.97 and 1.98.
14. ☐ An assignment document for recording. A separate cover sheet in compliance with 37 CFR 3.28 and 3.31 is included.
15. ☒ A **FIRST** preliminary amendment.
16. ☐ A **SECOND** or **SUBSEQUENT** preliminary amendment.
17. ☐ A substitute specification.
18. ☐ A change of power of attorney and/or address letter.
19. ☐ A computer-readable form of the sequence listing in accordance with PCT Rule 13ter.2 and 35 U.S.C. 1.821 - 1.825.
20. ☐ A second copy of the published international application under 35 U.S.C. 154(d)(4).
21. ☐ A second copy of the English language translation of the international application under 35 U.S.C. 154(d)(4).
22. ☐ Certificate of Mailing by Express Mail
23. ☒ Other items or information:

Copy of the application as published (WO 01/14136)
White Advance Serial Number Card

U.S. APPLICATION NO. (IF KNOWN, SEE 37 CFR	INTERNATIONAL APPLICATION NO	ATTORNEY'S DOCKET NUMBER
10/069130	PCT/EP00/08205	4004-030-30

24. The following fees are submitted:.		CALCULATIONS PTO USE ONLY	
BASIC NATIONAL FEE (37 CFR 1.492 (a) (1) - (5)) :			
☐ Neither international preliminary examination fee (37 CFR 1.482) nor international search fee (37 CFR 1.445(a)(2)) paid to USPTO and International Search Report not prepared by the EPO or JPO	**$1040.00**		
☒ International preliminary examination fee (37 CFR 1.482) not paid to USPTO but International Search Report prepared by the EPO or JPO	**$890.00**		
☐ International preliminary examination fee (37 CFR 1.482) not paid to USPTO but international search fee (37 CFR 1.445(a)(2)) paid to USPTO	**$740.00**		
☐ International preliminary examination fee (37 CFR 1.482) paid to USPTO but all claims did not satisfy provisions of PCT Article 33(1)-(4)	**$710.00**		
☐ International preliminary examination fee (37 CFR 1.482) paid to USPTO and all claims satisfied provisions of PCT Article 33(1)-(4)	**$100.00**		
ENTER APPROPRIATE BASIC FEE AMOUNT =		**$890.00**	
Surcharge of **$130.00** for furnishing the oath or declaration later than ☐ 20 ☒ 30 months from the earliest claimed priority date (37 CFR 1.492 (e)).		**$130.00**	

CLAIMS	NUMBER FILED	NUMBER EXTRA	RATE		
Total claims	20 - 20 =	0	x $18.00	**$0.00**	
Independent claims	2 - 3 =	0	x $84.00	**$0.00**	
Multiple Dependent Claims **(check if applicable)**.			☐	**$0.00**	
TOTAL OF ABOVE CALCULATIONS =				**$1,020.00**	
☐ Applicant claims small entity status. See 37 CFR 1.27). The fees indicated above are reduced by 1/2.				**$0.00**	
SUBTOTAL =				**$1,020.00**	
Processing fee of **$130.00** for furnishing the English translation later than ☐ 20 ☐ 30 months from the earliest claimed priority date (37 CFR 1.492 (f)). +				**$0.00**	
TOTAL NATIONAL FEE =				**$1,020.00**	
Fee for recording the enclosed assignment (37 CFR 1.21(h)). The assignment must be accompanied by an appropriate cover sheet (37 CFR 3.28, 3.31) **(check if applicable).** ☐				**$0.00**	
TOTAL FEES ENCLOSED =				**$1,020.00**	
				Amount to be: refunded	$
				charged	$

a. ☒ A check in the amount of **$1,020.00** to cover the above fees is enclosed.

b. ☐ Please charge my Deposit Account No. _____ in the amount of _____ to cover the above fees. A duplicate copy of this sheet is enclosed.

c. ☒ The Commissioner is hereby authorized to charge any additional fees which may be required, or credit any overpayment to Deposit Account No. **50-1442** A duplicate copy of this sheet is enclosed.

d. ☐ Fees are to be charged to a credit card. **WARNING:** Information on this form may become public. **Credit card information should not be included on this form.** Provide credit card information and authorization on PTO-2038.

NOTE: Where an appropriate time limit under 37 CFR 1.494 or 1.495 has not been met, a petition to revive (37 CFR 1.137(a) or (b)) must be filed and granted to restore the application to pending status.

SEND ALL CORRESPONDENCE TO:

Supervisor, Patent Prosecution Services
PIPER MARBURY RUDNICK & WOLFE, LLP
1200 Nineteenth Street, NW
Washington, DC 20036-2412
US

SIGNATURE

SCHNEIDER, Jerold I.
NAME

24,765
REGISTRATION NUMBER

22 FEBRUARY 2002
DATE

DOCKET NO. 4004-030-30

PATENT COOPERATION TREATY (PCT)

IN THE UNITED STATES DESIGNATED/ELECTED OFFICE (DO/EO/US)

In re Application of:	DEGAND, Etienne
Int'l Application No.:	PCT/EP00/08205
Int'l Filing Date:	21 August 2000
Priority Date:	26 August 1999
U.S. Application No.:	New U.S. National Stage Application
For:	**GLAZING**

PRELIMINARY AMENDMENT

Assistant Commissioner for Patents
Box PCT
Washington, D.C. 20231

SIR:

Prior to any examination on the merits, please amend this application as follows:

IN THE SPECIFICATION

Page 1, prior to line 3, insert the following including a section heading:

- - CROSS REFERENCE TO RELATED APPLICATIONS

This application is the U.S. National Stage of International Application No. PCT/EP00/08205 filed 21 August 2000, and European Application No. 99202757.3 filed 26 August 1999. The entirety of each of those applications is incorporated herein by reference.

BACKGROUND OF THE INVENTION - -

Page 4, between lines 11 and 12, insert the section heading:

- - SUMMARY OF THE INVENTION - -

Page 7, between lines 35 and 36, insert the section heading:

- - BRIEF DESCRIPTION OF THE FIGURES - -

Page 8, between lines 5 and 6, insert the section heading:

- - DETAILED DESCRIPTION - -

IN THE CLAIMS

Please cancel claims 1 – 29 without prejudice or disclaimer, and add the following new Claims 30 - 49.

- - 30. (new) A method of manufacturing a curved laminated automotive glazing panel, comprising the steps of:

(a) depositing a solar control coating layer comprising a coating stack having at least two spaced sputtered silver containing layers on a substantially flat sheet of glazing material;

(b) bending said substantially flat sheet of glazing material carrying said solar control coating layer such that the solar control coating layer is positioned at a convex surface of the bent sheet of glazing material;

(c) laminating said bent sheet of glazing material carrying the solar control coating layer at a convex surface with another sheet of glazing material to form a glazing panel in which the solar control coating layer is positioned at the interior of the glazing panel; and

wherein the laminated automotive glazing panel is curved according to at least one of the following (d) and (e):

(d) the curved laminated automotive glazing panel has at least one portion having a radius of curvature that is less than 500 mm;

(e) the curved laminated automotive glazing panel has a cross curvature of greater than or equal to 15 mm.

31. (new) A method according to Claim 30, wherein the laminated automotive glazing panel is curved according to both (d) and (e).

32. (new) A method according to Claim 30, and further including at least one of the following (f) through (m):

(f) the curved laminated automotive glazing panel has a depth of bending that is greater than or equal to 150 mm;

(g) the coating layer is adapted to be electrically heatable to provide a de-misting and/or de-icing function to the glazing panel and in which the glazing panel is provided with a pair of spaced bus bars adapted to relay electrical power to heat the solar control containing layer;

(h) the curved laminated automotive glazing panel has a width of greater than about 1.6 m;

(i) the curved laminated automotive glazing panel has a luminous transmittance of at least 75% (measured using Illuminant A, 2 degree observer);

(j) the colour co-ordinates of the curved laminated automotive glazing panel in reflection from the exterior measured on the CIElab scale at normal incidence are within the range:

$L^* = 40 \pm 3$ $a^* = -6 \pm 3$ $b^* = -8 \pm 4$; or

$L^* = 39 \pm 3$ $a^* = -6 \pm 3$ $b^* = -2 \pm 4$; or

$L^* = 36 \pm 3$ $a^* = -5 \pm 2$ $b^* = -4 \pm 2$;

(k) colour variation in reflection over the surface of the glazing panel is such that

when measured at different points over a single glazing, the values of a* and/or b* measured on the CIElab scale at normal incidence do not vary but more than ± 1.5;

(l) the electrical resistance of the coating layer is between 1.5 and 4 ohms per square; and

(m) the glazing panel is provided with a pair of spaced bus bars adapted to provide electrical power to heat the solar control coating layer and in which the resistance between the bus bars is between about 0.75 and 8 ohms.

33. (new) A method according to Claim 32, and including at least two of the aforementioned features (f) through (m).

34. (new) A method according to Claim 32, and including at least three of the aforementioned features (f) through (m).

35. (new) A method according to Claim 32, and including all of the aforementioned features (f) through (m).

36. (new) A method according to Claim 30, and including at least one of the following (n) through (p):

(n) the glazing panel has a radius of curvature at said at least one portion that is less than 400 mm;

(o) the glazing panel has a radius of curvature at said at least one portion that is less than 350 mm;

(p) the glazing panel has a radius of curvature at said at least one portion that is less than 300 mm.

37. (new) A method according to Claim 30 and including at least one of the following (q) through (s):

(q) the glazing panel has a cross curvature of greater than or equal to 20 mm;

(r) the glazing panel has a cross curvature of greater than or equal to 25 mm;

(s) the glazing panel has a cross curvature of greater than or equal to 30 mm.

38. (new) A method according to Claim 32, including the aforementioned (g) and wherein the glazing panel is provided with a substantially opaque band arranged at the internal, concave surface of the glazing panel adapted to mask the bus bars from view from the exterior of the glazing panel.

39. (new) A method according to Claim 30, in which the glazing panel is an automotive windscreen.

40. (new) A curved laminated automotive glazing panel, comprising:

(a) a solar control coating layer comprising a coating stack, said solar control coating layer positioned at the convex internal surface of the glazing panel;

(b) said coating stack having at least two spaced sputtered silver containing layers on a first substantially flat sheet of glazing material which is subsequently bent such that the solar control coating layer is at a convex surface of said first sheet of glazing material;

(c) another sheet of glazing material laminated to said first sheet of glazing material having said solar control coating layer positioned thereon, to form a glazing panel in which the solar control coating layer is positioned at the interior of the glazing panel; and

wherein the laminated automotive glazing panel is curved according to at least one of the following (d) and (e):

(d) the curved laminated automotive glazing panel has at least one portion having a radius of curvature that is less than 500 mm;

(e) the curved laminated automotive glazing panel has a cross curvature of greater than or equal to 15 mm.

41. (new) A glazing panel according to Claim 40, wherein the glazing panel is curved according to both (d) and (e).

42. (new) A glazing panel according to Claim 40, and further including at least one of the following (f) through (m):

(f) the curved laminated automotive glazing panel has a depth of bending that is greater than or equal to 150 mm;

(g) the coating layer is adapted to be electrically heatable to provide a de-misting and/or de-icing function to the glazing panel and in which the glazing panel is provided with a pair of spaced bus bars adapted to relay electrical power to heat the solar control containing layer;

(h) the curved laminated automotive glazing panel has a width of greater than about 1.6 m;

(i) the curved laminated automotive glazing panel has a luminous transmittance of at least 75% (measured using Illuminant A, 2 degree observer);

(j) the colour co-ordinates of the curved laminated automotive glazing panel in reflection from the exterior measured on the CIElab scale at normal incidence are within the range:

$L^* = 40 \pm 3$ $a^* = -6 \pm 3$ $b^* = -8 \pm 4$; or

$L^* = 39 \pm 3$ $a^* = -6 \pm 3$ $b^* = -2 \pm 4$; or

$L^* = 36 \pm 3$ $a^* = -5 \pm 2$ $b^* = -4 \pm 2$;

(k) colour variation in reflection over the surface of the glazing panel is such that when measured at different points over a single glazing, the values of a* and/or b* measured on the CIElab scale at normal incidence do not vary but more than ± 1.5;

(l) the electrical resistance of the coating layer is between 1.5 and 4 ohms per square; and

(m) the glazing panel is provided with a pair of spaced bus bars adapted to provide electrical power to heat the solar control coating layer and in which the resistance between the bus bars is between about 0.75 and 8 ohms.

43. (new) A glazing panel according to Claim 42, and including at least two of the aforementioned features (f) through (m).

44. (new) A glazing panel according to Claim 42, and including at least three of the aforementioned features (f) through (m).

45. (new) A glazing panel according to Claim 42, and including all of the aforementioned features (f) through (m).

46. (new) A glazing panel according to Claim 41, and including at least one of the following (n) through (p);

(n) the glazing panel has a radius of curvature at said at least one portion that is less than 400 mm;

(o) the glazing panel has a radius of curvature at said at least one portion that is less than 350 mm;

(p) the glazing panel has a radius of curvature at said at least one portion that is less than 300 mm.

47. (new) A glazing panel according to Claim 41, and including at least one of the following (q) through (s):

(q) the glazing panel has a cross curvature of greater than or equal to 20 mm;

(r) the glazing panel has a cross curvature of greater than or equal to 25 mm;

(s) the glazing panel has a cross curvature of greater than or equal to 30 mm.

48. (new) A glazing panel according to Claim 42, including the aforementioned (g) and wherein the glazing panel is provided with a substantially opaque band arranged at the internal, concave surface of the glazing panel adapted to mask the bus bars from view from the exterior of the glazing panel.

49. (new) A glazing panel according to Claim 40, in which the glazing panel is an automotive windscreen. - -

REMARKS

This is the entry into the U.S. National Stage of International Application No. PCT/EP00/08205 filed 21 August 2000. The foregoing amendments place the specification into U.S. format and present Claims 30 – 49 for examination.

Applicant respectfully submits that, in view of the foregoing amendments and remarks, the application is in condition for examination. Favorable consideration is respectfully requested.

Respectfully submitted,

PIPER MARBURY RUDNICK & WOLFE LLP

Jerold I. Schneider
Registration No. 24,765

1200 Nineteenth Street, N.W.
Washington, D.C. 20036-2412
Telephone No. (202) 861-3900
Facsimile No. (202) 223-2085

2/prts

Glazing

This invention relates to laminated automotive glazing panels provided with solar control coating layers. Whilst the invention will be particularly described with reference to vehicle windscreens it should be understood that it may have other applications.

The use of coating layers is well know to modify the optical properties of glazings. In particular, coating layers may be used to reduce the proportion of incident solar energy which is transmitted through the glazing whilst allowing passage of sufficient visible light to ensure good visibility. This can reduce overheating of the interior of the vehicle in summer and is commonly achieved by reflection of incident solar radiation in the infra-red portion of the spectrum. Coating layers may also provide an electrically heatable element for a glazing. EP378917A (Nippon Sheet Glass Co.) discloses such coating layers.

The term solar control coating layer as used herein refers to a coating layer which increases the selectivity of the glazing panel i.e. the ratio of the proportion of incident visible radiation transmitted through the glazing to the proportion of incident solar energy transmitted through the glazing.

The term luminous transmittance as used herein means the luminous flux transmitted through a substrate as a percentage of the incident luminous flux measured using CIE Illuminant A at 2° observer.

There are a number of different families of solar control coating layers, each of which have differing properties and characteristics. These include:

a) pyrolytic coatings obtained by contacting a liquid or vapour composition with the hot surface of a glass sheet. Such coatings include tin oxide coatings doped with fluorine and indium tin oxide (ITO) coatings. Pyrolytic coatings have the general characteristic of being hard coatings (i.e. they are relatively resistant to abrasion) and of being relatively easy to handle during manufacturing processes without damage to the coating layer. Many pyrolytic coatings are inherently heat resistant to a sufficient extent to enable glass sheets to which they are applied to be bent and/or thermally tempered without significant deterioration of their solar control properties. A significant industrial advantage results from the ability to apply pyrolytic coatings to a continuous ribbon of flat glass for example as part of a process of making float glass. European patent application EP 353 141 A (Saint Gobain Vitrage) describes a heatable pyrolytic indium tin oxide coating on face 3 of a laminated windscreen. Such glazings have never found commercial success

in the automotive field due particularly to the inherent optical and energetic limitations of this type of coating.

b) Sputtered single silver layer coatings, obtained by sputtering a silver containing layer on to a supporting substrate. Such coatings usually comprise a coating stack having the general form: supporting substrate/ antireflective layer/ optional barrier layer/ silver containing layer/ optional barrier/ antireflective layer. In such a structure the silver containing layer serves to reflect radiation in the infra red portion of the spectrum, the antireflective layers serve to reduce reflection of light in the visible portion of the spectrum that would otherwise be caused by the silver containing layer and the optional barrier layers serve to protect to silver containing layer either during deposition of the coating and/or subsequent processing. Whilst the optical performance of single silver layer sputtered coatings is reasonably good such sputtered coatings are generally "soft" coatings i.e. they are not particularly resistant to abrasions and scratches and require significant care in handling to avoid damage. In addition, significant care in both the design and handling of such layers is required to enable them to be sufficiently heat resistant to allow tempering and/or bending of a substrate to which they are applied.

c) Sputtered double silver layers obtained by sputtering two, spaced silver layers onto a supporting substrate. Such coatings usually comprise a coating stack having the general form: supporting substrate/ antireflective layer/ optional barrier layer/ silver containing layer/ optional barrier layer/ antireflective layer/ optional barrier layer/ silver containing layer/ optional barrier/ antireflective layer. In such a structure the silver containing layers serve to reflect radiation in the infra red portion of the spectrum, the antireflective layers serve to reduce reflection of light in the visible portion of the spectrum that would otherwise be caused by the silver containing layer and the optional barrier layers serve to protect to silver containing layers either during deposition of the coating and/or subsequent processing. The infra red reflective silver containing layers are commonly layers of silver or a silver alloy have a thickness in the order of 80 to 120 Å. The optical performance of double silver layer sputtered coatings can be extremely good, especially in terms of their selectivity but perhaps even more so than with single silver sputtered coatings these coatings are extremely fragile both in terms of resistance to abrasions and scratches (for example during handling) and in their ability to withstand heating for example to enable them to be sufficiently heat resistant to allow tempering and/or bending of a substrate to which they are applied.

One example of the use of sputtered coating layers in automotive applications is US Patent N° 4,668,270 (Ford Motor Company) which describes a car

windscreen having an electrically heatable coating layer used for defrosting, de-icing and/or de-misting. The heatable coating, which is laminated between the two glass sheets of the windscreen, is supplied with electrical power via first and second bus bars which extend respectively along the top and bottom edges of the windscreen, each bus bar being silk screen printed on the glass in a silver ceramic material. The heatable coating is a multilayer coating consisting of zinc oxide and silver formed by magnetron sputtering.

The physical nature of double silver layer coatings layers is entirely different to that of, for example, pyrolytic coating layers and, consequently, entirely different techniques must be employed for their design, processing and use.

It has generally been believed in the art that the precautions of the techniques described below must be adhered to to enable the successful use of sputtered double silver layers in laminated automotive glazings:

1) that the sputtered double silver layers should be deposited on a carrier film of, for example pet (poly ethylene tetrachloride), which is assembled between the two glass sheets of a laminated glazing once the individual sheets have been bent to their desired final shape. One disadvantage of such carrier films is the difficulty of ensuring that the film correctly follows the precise contour of the bent glazing panel. Consequently, this procedure is limited to use with glazing panels of a relatively simple curvature. In addition, it is generally not desirable to electrically heat the solar control coating in such an arrangement due to deterioration of the coating and/or of the carrier film and it is also inconvenient to provide bus bars in this arrangement to relay electrical power to the coating. Consequently, this technique is generally unsuitable for use with heatable windscreens.

2) that, alternatively, the sputtered double silver layers should be applied to the concave face of a pre-bent sheet of glass prior to its assembly to form a laminated glazing panel. In this way, the coating layer is not subjected to the heat treatment necessary to form the desired curvature of the glass sheet. Disadvantages of this technique include the technical difficulty of sputter depositing coating layers onto a curved sheet of glass so as to ensure that the entire glass surface is evenly coated (due, amongst other things, to the variation in the distance between the different parts of the glazing surface and the targets used for the sputtering process - small variations in thickness of the coating layers can cause undesirable colour variations across the glazing panel) and the complexity and limitations (including dimensional limitations- complex windscreens having deep curvatures will not always fit in to such coating machines simply because of their dimensions) of coaters which can sputter deposit layers onto a curved substrate. Consequently,

this technique is also limited to use with relatively simple curvatures of glazing panels.

3) alternatively, sputter depositing a double silver coating layer onto a relatively flat sheet of glass and subsequently bending the glass sheet carrying the coating stack to its desired shape prior to assembly as a laminated glazing panel. Due to the fragility of this type of coating, the glass sheet carrying the coating stack should be bent such that the coating stack is at the concave face of the curved sheet of glass. This is so that the layers of the coating stack have a tendency to be compressed during the bending process so as to ensure the integrity and continuity of the layers of the coating stack; this is particularly so for complex curvatures of glazing panels.

Thus, in order to successfully use a sputtered double silver layer coating on a complex curved glazing panel it is necessary either to deposit the coating onto a pre-curved glazing panel or to use a heat treatable sputtered double silver layer coating deposited on a substantially flat sheet of glass which is subsequently bent so that the coating is at its concave surface.

According to one aspect, the present invention provides a method of making a laminated automotive glazing panel as defined in Claim 1.

The invention results from the unexpected realisation that despite prejudice in the art, a complex shape of glazing panel incorporating a sputtered double silver layer coating may be produced on an industrial scale by depositing the coating on a substantially flat sheet of glass and subsequently bending the glass sheet carrying the coating such that the coating is at the convex surface of the glazing panel. This is possible despite the fact that subjecting the sputtered double silver coating layer to the tension and extension inherent in bending it into a convex shape would be expected to destroy the integrity and continuity of the coating layer and perhaps even to result in significant disparities in the thickness of the coating layer over the area of the glazing panel. The realisation that this is possible with relatively complex curvatures is even more surprising firstly because complex curvatures require significant degrees of bending and would thus be expected to cause unacceptable tension and extension of the coating layer and secondly because complex curvatures require the heating of the glass substrate to a softening level at higher temperature and/or for a longer duration that simple curvatures and would thus be expected to put additional unacceptable strains on the relatively fragile sputtered double silver coating layer.

One factor which may be used to define the complexity of curvature of an automotive glazing is the radius of curvature. The smaller the radius of curvature, the more difficult it becomes to accurately and repeatably bend the glazing panel.

The present invention may be used in association with glazing panels having a radius of curvature at at least one portion that is less than 450mm, less than 400mm, less than 350mm, less than 300 mm, less than 250mm, less than 200mm , less than 150mm or even less.

According to another aspect, the present invention provides a method of making a laminated automotive glazing panel as defined in Claim 3.

Another factor that may be used to define the complexity of curvature of an automotive glazing panel is its cross curvature. This is a measurement of the depth of curvature across the height of the glazing at the central portion of the glazing panel.

The present invention may be used in association with glazing panels having a cross curvature that is at least 15mm, at least 20mm, at least 25mm, at least 30mm, at least 35mm or greater.

The curvature of the glazing panel becomes even more complex when, for example, a significant minimum radius is combined with a significant cross curvature.

A further factor which may add to the complexity of curvature of an automotive glazing panel is the depth of bending. This is a measure of the greatest distance between the front face of the glazing panel and the end of the rearwardly projecting side wings of the glazing panel. The present invention may be used in association with a depth of bending of at least 150mm, at least 170mm, at least 190mm, at least 200mm, at least 220mm, at least 240 mm, at least 250mm or more. The complexity of curvature is further increased if a significant depth of bending is combined with a significant minimum radius of curvature and/or a significant cross curvature.

The present invention may advantageously be used to provide a de-misting and/or de-icing function to the glazing by using a coating layer which is electrically heatable and providing a pair of spaced bus bars to relay electrical current to the coating layer. The exposed concave surface of a laminated windscreen is generally at the interior of the vehicle. Positioning the coating layer on the convex surface sandwiched between the two glazing panels of the laminated structure may provide a number of advantages with respect to positioning a heatable sputtered double silver coating layer at the concave surface sandwiched between the two glazing panels of the laminated structure. These may include:

a) an improved de-misting function as the heatable coating layer is directly adjacent to the sheet of the glazing panel at the interior of the vehicle;

b) a reduced risk of damaging the integrity of the coating layer in the case of a small breakage or crack of the outer sheet of the glazing panel, for example due to the impact of gravel. Any discontinuity in the coating layer may cause a break in its electrical conductivity and a consequent overheating of immediately surrounding areas of the coating layer when electrical current passes. Such overheating may cause deterioration of the coating layer and/or deterioration of the laminating film between the two sheets of the glazing panel. In addition, the position of the coating protects it at least to some degree from the risk of corrosion by the ingress of moisture in the case of a breakage or crack in the outer sheet of the glazing panel.

An additional advantage of the defined positioning of the coating layer in association with the provision of bus bars is the ability the hide the bus bars from view from the exterior of the glazing panel by providing a substantially opaque masking layer, for example a black enamel layer, around a portion of the internal, concave surface of the glazing panel.

The complexity of curvature is increased in respect of glazing panels having a significant width, for example, glazing panels that are between 1.2m and 1.4m wide, or between 1.4m and 1.6m wide or between 1.6m and 1.8m wide.

The invention may be particularly suitable for use in relation to vehicle windscreens.

The glazing panel may have a luminous transmittance of greater than 70% or greater than 75%. This may enable its use as a windscreen.

The glazing panel may have a neutral colour in reflection from its exterior surface, a slightly blue colour or a slightly green colour. This may render it particularly suitable for use in automotive applications. In particular, the colour of the glazing panel in reflection from the exterior may be such that its colour co-ordinates measured on the CIElab scale at normal incidence are:

$L^*=40 \pm 3$ $a^*=-6 \pm 3$ $b^*=-8 \pm 4$ (this is intended to give a blue tint in reflection, particularly for a windscreen installed at an angle); or

$L^*=39 \pm 3$ $a^*=-6 \pm 3$ $b^*=-2 \pm 2$ (this is intended to give a green tint in reflection, particularly for a windscreen installed at an angle); or

$L^*=36 \pm 3$ $a^*=-5 \pm 2$ $b^*=-4 \pm 2$ (this is intended to give a neutral/green tint in reflection, particularly for a windscreen installed at an angle);

The variation is colour in reflection over the surface of the glazing panel may be such that when measured at different points over a single glazing, the

values of either a* and/or b* measured on the CIElab scale at normal incidence do not vary by more than ±1.5, and preferably by not more than ±1. The variation in colour in reflection is due at least to a significant extent upon variations in the thickness of the film stacks of the coating layer and/or variations in the heating regime during heat treatment over different parts of the glazing. It is perhaps particularly unexpected that such minimal colour variation can be achieved by means of the present invention as it would be expected that extension of the coating layer would stretch the coating layer, at least in some places and/or destroy the integrity of the coating layer and/or render it unstable during heat treatment.

Preferably, the variation in colour in reflection between one glazing and another is such that the values of either a* and/or b* measured on the CIElab scale at normal incidence do not vary between one glazing and another in a series of glazings by more than ±2, and preferably by not more than ±1.5.

Arranging the resistance of the heatable coating layer to be between about 1.5 and 4 ohms per square may provide particularly suitable heating characteristics for automotive use. Similarly, arranging for the resistance between the bus bars to be between 0.75 ohm and 8 ohm may also provide particularly suitable heating characteristics for automotive use.

According to further aspects, the present invention also provides a curved laminated glazing panel, as defined in claims 15 and 17, and for the use of a sputtered double silver coating layer deposited on a substantially flat sheet of glazing material and subsequently bent into a convex configuration to provide a glazing panel as defined in claim 29.

The glazing material onto which the solar control coating layer is deposited may be a sheet of glass. It is preferably a soda-lime glass, more preferably float glass. It may comprise the following constituent (expressed in percentage by weight):

SiO_2	60 to 75%
Na_2O	10 to 20%
CaO	0 to 16%
K_2O	0 to 10%
MgO	0 to 10%
Al_2O_3	0 to 5%
BaO	0 to 2%
BaO+CaO+MgO	10 to 20%
K_2O+Na_2O	10 to 20%

An embodiment of the present invention will now be described, by way of example only, with reference to:

Fig. 1 which is an exploded view showing the overall structure (but not the curvature) of a windscreen;

Fig 2 which is a plan view of a curved, laminated, automotive windscreen;

Fig 3 which is a section along line 3-3 of Fig 2; and

Fig 4 which is a section along line 4-4 of Fig 2.

Windscreen 10 illustrated in Fig 1 comprises an inner sheet of glass 11 laminated to an outer sheet of glass 13 by means of a sheet of pvb 12.

The windscreen is substantially trapezial in shape having a top edge 21, a longer bottom edge 23 substantially parallel thereto and side edges 22, 24. The windscreen has a spherical, curved configuration so that it is curved both along an axis parallel to the top edge 21 and along an axis perpendicular to the top edge 21 (for ease of representation the curvature of the windscreen is not shown in Fig 1).

An electrically conducting solar control layer 25 comprising a sputtered double silver coating layer is positioned on the convex face of the inner glazing sheet 11 between the inner and outer sheets of glass 11,13.

The coating layer 25 is produced by sputtering the following layers sequentially onto a substantially flat sheet of glass which is subsequently bent to form the inner glazing sheet 11:

	Geometrical thickness	Atomic ratios
Glass substrate	2 mm	
Base dielectric comprising: AlN ZnAlOx	60 Å 250 Å	Al/Zn=0.1
ZnAlOy underlying barrier	10 Å	Al/Zn=0.1
Ag	100 Å	
ZnAlOy overlying barrier	12 Å	Al/Zn=0.1
Central dielectric comprising ZnAlOx	750 Å	Al/Zn=0.1
ZnAlOy underlying barrier	7 Å	Al/Zn=0.1
Ag	100 Å	
ZnAlOy overlying barrier	17 Å	Al/Zn=0.1
Top dielectric comprising: ZnAlOx AlN	 185 Å 85 Å	Al/Zn=0.1

in which ZnAlOx is a mixed oxide containing Zn and Al deposited in this example by reactively sputtering a target which is an alloy or mixture of Zn and Al in the presence of oxygen .

Alternatively, a mixed oxide layer may be formed by sputtering a target which is a mixture of zinc oxide and aluminium oxide particularly in an argon gas or argon rich oxygen containing atmosphere.

The ZnAlOy barriers are similarly deposited by sputtering a target which is an alloy or mixture of Zn and Al in an argon rich oxygen containing atmosphere to deposit a barrier that is not fully oxidised.

The oxidation state in each of the base, central and top ZnAlOx dielectric layers need not necessarily be the same. Similarly, the oxidation state in each of the ZnAlOy barriers need not be the same. Equally, the Al/Zn ratio need not be the same for all of the layers; for example, the barrier layers may have a different Al/Zn ratio to the antireflective dielectric layers and the antireflective dielectric layers may have different Al/Zn ratios from each other.

Each overlying barrier protects its underlying silver layer from oxidation during sputter deposition of its overlying ZnAlOx oxide layer. Whilst further oxidation of these barriers layers may occur during deposition of their overlying oxide layers a portion of these barriers preferably remains in the form of an oxide that is not fully oxidised to provide a barrier for subsequent heat treatment of the glazing panel.

The glazing sheet carrying the sputtered double silver coating stack is subsequently heated, bent to its desired curvature and assembled with a sheet of pvb into a laminated vehicle windscreen which has the following properties:

Property	Prior to heat treatment see Note 1 below	Following heat treatment see Note 2 below
TL(Illuminant A)	63%	76%
TE (System Moon 2)	38%	42%
haze	0.1	0.25
a*	-20 (coated side)	-6 (external)
b*	+3 (coated side)	-12(external)
RE (System Moon 2)	31% (coated side)	33%(external)

Note 1: Measured for monolithic glazing panel with coating prior to heat treatment

Note 2: Measured following heat treatment at 650° C for 10 minutes followed by bending and tempering, and lamination with clear 2mm glass sheet and 0.76mm clear pvb

Heat treatment preferably causes substantially complete oxidation of all of the barrier layers such that the structure of the coating stack after heat treatment is:

	Geometrical thickness	Atomic ratios
Glass substrate	2 mm	
Base dielectric comprising: AlN (partially oxidised) ZnAlOx	 60 Å 250 Å	 Al/Zn=0.1
ZnAlOx (oxidised underlying barrier)	10 Å - 16 Å	Al/Zn=0.1
Ag	100 Å	
ZnAlOx (oxidised overlying barrier)	12 Å -20 Å	Al/Zn=0.1
Central dielectric comprising ZnAlOx	750 Å	Al/Zn=0.1
ZnAlOx (oxidised underlying barrier)	7 Å -12 Å	Al/Zn=0.1
Ag	100 Å	
ZnAlOx (oxidised overlying barrier)	17 Å - 28 Å	Al/Zn=0.1
Top dielectric comprising: ZnAlOx AlN (partially oxidised)	 185 Å 85 Å	 Al/Zn=0.1

The AlN (partially oxidised) layers may comprise a mixture of AlN and Al_2O_3, the AlN being partially oxidised during the heat treatment process. The barrier layers are not necessarily completely oxidised and their thickness will depend to a certain extent upon their degree of oxidation.

The coating layer 25 is spaced from the external periphery of the windscreen by a non-conducting peripheral band (not shown) provided in this example by a band in which the coating layer has either not been deposited or has been removed. This prevents the electrically conductive coating extending to the

very edge of the windscreen and may also reduce the risk of corrosion of the coating layer.

Electrical power is supplied to the coating layer via a first bus bar 31 arranged in contact with the coating layer 25 adjacent to the top edge 21 of the windscreen and a second bus bar 32 arranged in contact with the coating layer 25 adjacent to the bottom edge 23 of the windscreen. Connectors 33,34 for facilitating connection of the bus bars to a car's electrical circuit may protrude from the glazing and may be arranged adjacent to each other. The first bus bar has a portion which runs down the side edge 22 of the windscreen along a portion of the glazing panel 11 at which the coating layer 25 is not present so that there is no electrical connection between this portion of the bus bar and the coating layer. This allows the connector 33 to be positioned at the bottom edge 22 of the windscreen. The bus bars may be formed in any suitable manner, for example by silk screen printing of a conducting enamel material underneath or on top of the coating layer or by means of conducting tape or metal strips.

Figs 2,3 and 4 illustrate the curvature and dimensions of the glazing, the significant measurements in this case being:

w the width of the glazing window

r the radius of curvature of the glazing panel. Different portions of the glazing panel will have different radii of curvature

cc the cross curvature of the windscreen. The cross curvature may be measured by placing the convex surface 13 of the windscreen on a surface, placing a straight, rigid bar at the concave face of the windscreen along axis y such that the bar rests against a point at the top edge 21 of the windscreen and against a point at the bottom edge 23 of the windscreen and measuring the distance between the underside of the bar and the concave surface of the windscreen. The maximum cross curvature is the maximum distance, usually at the centre of the windscreen, which the windscreen has been bent parallel to axis y.

d the depth of the windscreen. The maximum depth is the maximum distance measured parallel to axis z between the outer surface of the convex surface of the laminated windscreen 13 and the furthest spaced portion on the side wings of the windscreen.

Claims

1. A method of manufacturing a curved laminated automotive glazing panel having a radius of curvature at at least one portion that is less than 500 mm comprising the steps of:

a) depositing a solar control coating layer comprising a coating stack having at least two spaced sputtered silver containing layers on a substantially flat sheet of glazing material;

b) bending said substantially flat sheet of glazing material carrying said solar control coating layer such that the solar control coating layer is positioned at a convex surface of the bent sheet of glazing material;

c) laminating said bent sheet of glazing material carrying the solar control coating layer at a convex surface with another sheet of glazing material to form a glazing panel in which the solar control coating layer is positioned at the interior of the laminated glazing panel.

2. A method of manufacturing a curved laminated automotive glazing panel in accordance with claim 1, in which the glazing panel has a radius of curvature at at least one portion that is less than 400 mm, preferably less than 350mm and even more preferably less than 300 mm.

3. A method of manufacturing a curved laminated automotive glazing panel having a cross curvature of greater than or equal to 15 mm comprising the steps of:

a) depositing a solar control coating layer comprising a coating stack having at least two spaced sputtered silver containing layers on a substantially flat sheet of glazing material;

b) bending said substantially flat sheet of glazing material carrying said solar control coating layer such that the solar control coating layer is positioned at a convex surface of the bent sheet of glazing material;

c) laminating said bent sheet of glazing material carrying the solar control coating layer at a convex surface with another sheet of glazing material to form a glazing panel in which the solar control coating layer is positioned at the interior of the glazing panel.

4. A method of manufacturing a curved laminated automotive glazing panel in accordance with any preceding claim, in which the glazing panel has a cross

curvature of greater than or equal to 20 mm, preferably greater than or equal to 25 mm and even more preferably greater than or equal to 30 mm.

5. A method of manufacturing a curved laminated automotive glazing panel in accordance with any preceding claim, in which the glazing panel has a depth of bending that is greater than or equal to 150 mm.

6. A method of manufacturing a curved laminated automotive glazing panel in accordance with any preceding claim, in which the coating layer is adapted to be electrically heatable to provide a de-misting and/or de-icing function to the glazing panel and in which the glazing panel is provided with a pair of spaced bus bars adapted to relay electrical power to heat the solar control coating layer.

7. A method of manufacturing a curved laminated automotive glazing panel in accordance with claim 6, in which the glazing panel is provided with a substantially opaque band arranged at the internal, concave surface of the glazing panel adapted to mask the bus bars from view from the exterior of the glazing panel.

8. A method of manufacturing a curved laminated automotive glazing panel in accordance with any preceding claim, in which the glazing panel has a width of greater than about 1.6 m.

9. A method of manufacturing a curved laminated automotive glazing panel in accordance with any preceding claim, in which the glazing panel is an automotive windscreen.

10. A method of manufacturing a curved laminated automotive glazing panel in accordance with any preceding claim, in which the glazing panel has a luminous transmittance of at least 75% (measured using Illuminant A, 2 degree observer).

11. A method of manufacturing a curved laminated automotive glazing panel in accordance with any preceding claim, in which the colour co-ordinates of the glazing panel in reflection from the exterior measured on the CIElab scale at normal incidence are within the range:

L*=40 ±3 a*=-6 ±3 b*=-8 ±4; or
L*=39 ±3 a*=-6 ±3 b*=-2 ±2; or
L*=36 ±3 a*=-5 ±2 b*=-4 ±2 .

12. A method of manufacturing a curved laminated automotive glazing panel in accordance with any preceding claim, in which colour variation in reflection over the surface of the glazing panel is such that when measured at different points over a single glazing, the values of either a* and/or b* measured on the CIElab scale at normal incidence do not vary by more than ±1.5, and preferably by not more than ±1.

13. A method of manufacturing a curved laminated automotive glazing panel in accordance with any preceding claim, in which the electrical resistance of the heatable coating layer is between 1.5 and 4 ohms per square.

14. A method of manufacturing a curved laminated automotive glazing panel in accordance with any preceding claim, in which the glazing panel is provided with a pair of spaced bus bars adapted to provide electrical power to heat the solar control coating layer and in which the resistance between the bus bars is between about 0.75 and 8 ohms.

15. A curved laminated automotive glazing panel having a radius of curvature at at least one portion that is less than 500 mm in which the glazing panel is provided with a solar control coating layer positioned at its convex internal surface and in which the coating stack comprises at least two spaced sputtered silver containing layers initially deposited on a substantially flat sheet of glazing material which is subsequently bent to form a part of the glazing panel.

16. A curved laminated automotive glazing panel in accordance with claim 15, in which the glazing panel has a radius of curvature at at least one portion that is less than 400 mm, preferably less than 350mm and even more preferably less than 300 mm.

17. A curved laminated automotive glazing panel having a cross curvature of greater than or equal to 15 mm in which the glazing panel is provided with a solar control coating layer positioned at its convex internal surface and in

which the coating stack comprises at least two spaced sputtered silver containing layers initially deposited on a substantially flat sheet of glazing material which is subsequently bent to form a part of the glazing panel.

18. A curved laminated automotive glazing panel in accordance with any one of claims 15 to 17, in which the glazing panel has a cross curvature of greater than or equal to 20 mm, preferably greater than or equal to 25 mm and even more preferably greater than or equal to 30 mm.

19. A curved laminated automotive glazing panel in accordance with any one of claims 15 to 18, in which the glazing panel has a depth of bending that is greater than or equal to 150 mm.

20. A curved laminated automotive glazing panel in accordance with any one of claims 15 to 19, in which the coating layer is adapted to be electrically heatable to provide a de-misting and/or de-icing function to the glazing panel and in which the glazing panel is provided with a pair of spaced bus bars adapted to relay electrical power to heat the solar control coating layer.

21. A curved laminated automotive glazing panel in accordance with claim 20, in which the glazing panel is provided with a substantially opaque band arranged at the internal, concave surface of the glazing panel adapted to mask the bus bars from view from the exterior of the glazing panel.

22. A curved laminated automotive glazing panel in accordance with any one of claims 15 to 21, in which the glazing panel has a width of greater than about 1.6 m.

23. A curved laminated automotive glazing panel in accordance any one of claims 15 to 22, in which the glazing panel is an automotive windscreen.

24. A curved laminated automotive glazing panel in accordance any one of claims 15 to 23, in which the glazing panel has a luminous transmittance of at least 75% (measured using Illuminant A, 2 degree observer).

25. A curved laminated automotive glazing panel in accordance any one of claims 15 to 24, in which the colour of the glazing panel in reflection from

the exterior is such that the colour co-ordinates of the glazing panel in reflection from the exterior measured on the CIElab scale at normal incidence are within the range:
L*=40 ±3 a*=-6 ±3 b*=-8 ±4; or
L*=39 ±3 a*=-6 ±3 b*=-2 ±2; or
L*=36 ±3 a*=-5 ±2 b*=-4 ±2 .

26. A curved laminated automotive glazing panel in accordance any one of claims 15 to 25, in which colour variation in reflection over the surface of the glazing panel is such that when measured at different points over a single glazing, the values of either a* and/or b* measured on the CIElab scale at normal incidence do not vary by more than ±1.5, and preferably by not more than ±1.

27. A curved laminated automotive glazing panel in accordance any one of claims 15 to 26, in which the electrical resistance of the heatable coating layer is between 1.5 and 4 ohms per square.

28. A curved laminated automotive glazing panel in accordance any one of claims 15 to 27, in which the glazing panel is provided with a pair of spaced bus bars adapted to provide electrical power to heat the solar control coating layer and in which the resistance between the bus bars is between 0.75 and 8 ohms.

29. Use of a sputter deposited double silver coating layer which is initially deposited on a substantially flat glazing sheet and subsequently bent into a convex configuration to provide a laminated automotive glazing panel in accordance with any one of claims 15 to 28.

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(71) Applicant *(for all designated States except US)*: **GLAVERBEL** [BE/BE]; Chaussée de La Hulpe, 166, B-1170 Brussels (Watermael-Boitsfort) (BE).

(72) Inventor; and
(75) Inventor/Applicant *(for US only)*: **DEGAND, Etienne** [BE/BE]; Glaverbel - Centre R. & D., Rue de l'Aurore, 2, B-6040 Jumet (BE).

(74) Agents: **FARMER, Guy** et al.; Glaverbel, Department Intellectual Property, Centre R. & D., Rue de l'Aurore, 2, B-6040 Jumet (BE).

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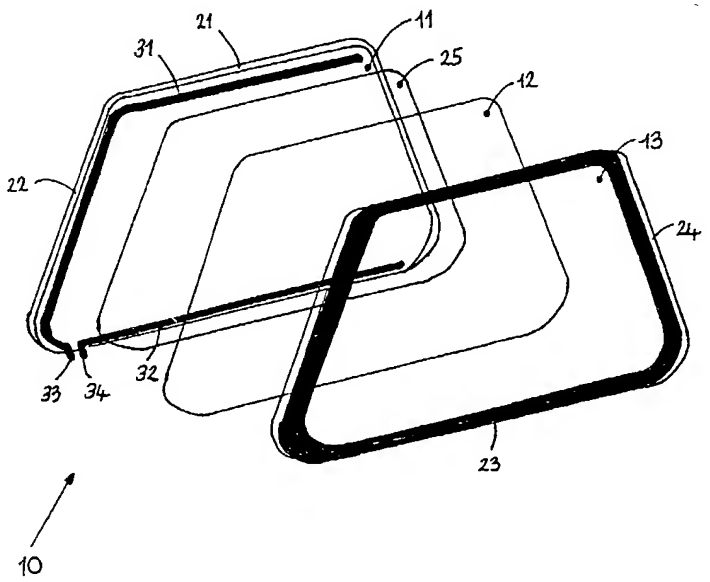

(57) Abstract: A curved laminated automotive glazing panel (10) having a radius of curvature at at least one portion that is less than 500 mm has a glazing panel which is provided with a solar control coating layer (25) positioned at its convex internal surface (11) and in which the coating stack comprises at least two spaced sputtered silver containing layers initially deposited on a substantially flat sheet of glazing material which is subsequently bent to form a part of the glazing panel (10).

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FIG 1

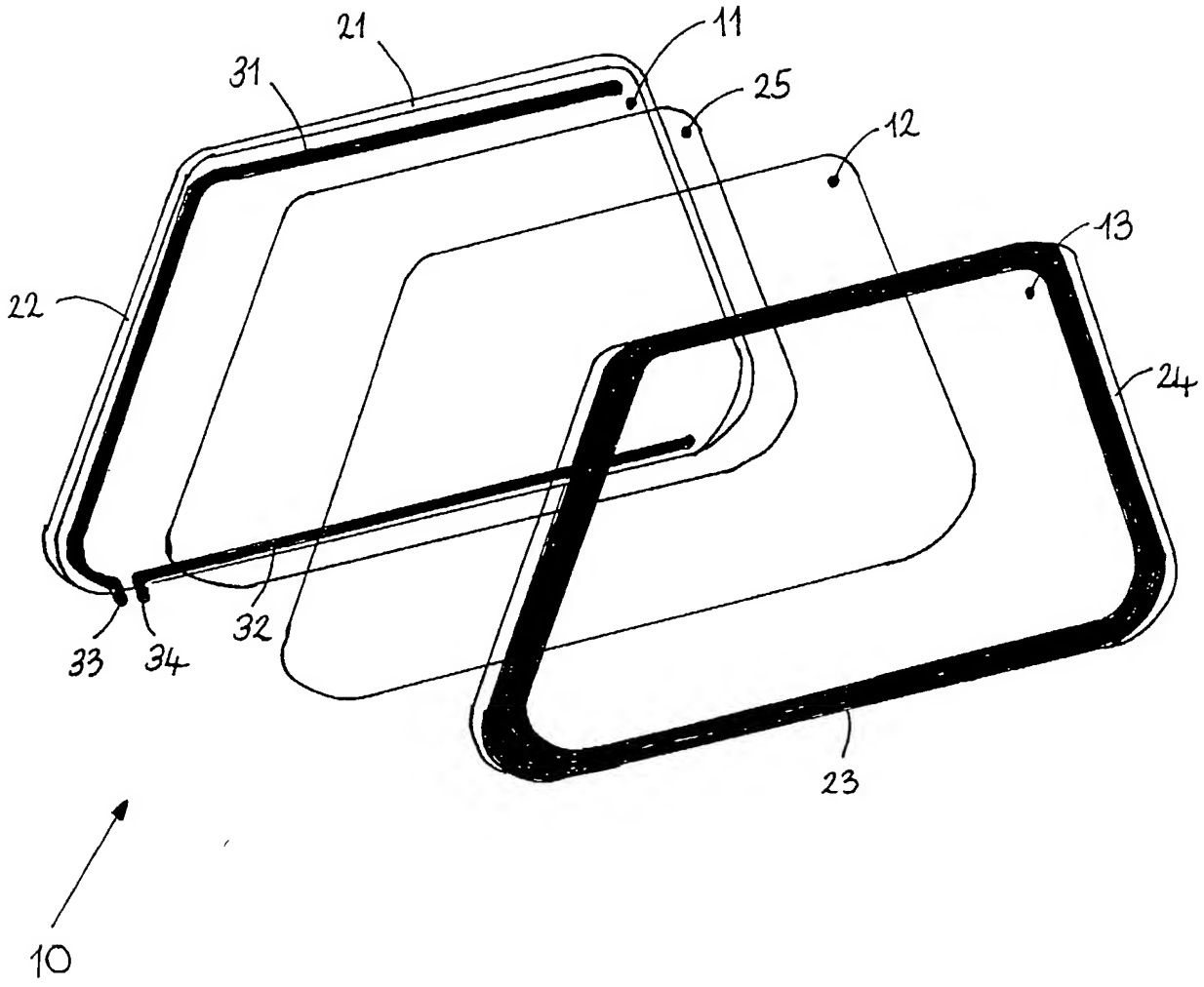

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FIG 2

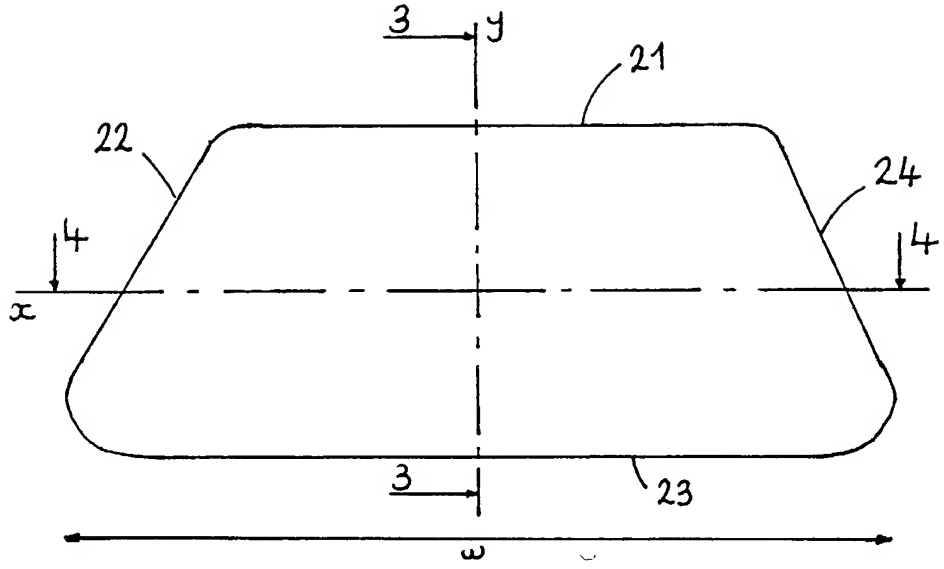
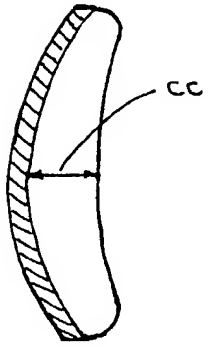


FIG 3



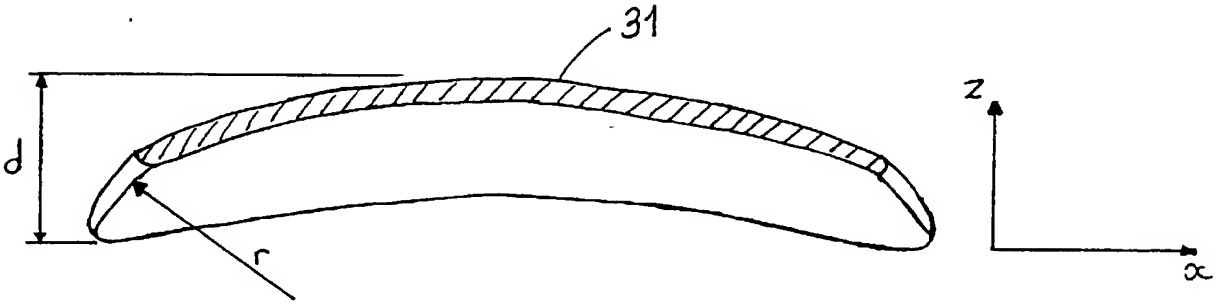


FIG 4

Docket No.: 4004-030-30

Declaration, Power of Attorney and Petition

WE (I) the undersigned inventor(s), hereby declare(s) that:

My residence, post office address and citizenship are as stated below next to my name,

We (I) believe that we are (I am) the original, first, and joint (sole) inventor(s) of the subject matter which is claimed and for which a patent is sought on the invention entitled

GLAZING

the specification of which

- ☐ is attached hereto.
- ■ was amended on 22 FEBRUARY 2002
- ■ was filed as PCT international application

Number PCT/EP00/08205

on 21 AUGUST 2000

and was amended under PCT Article 19

on _____ (if applicable).

We (I) hereby state that we (I) have reviewed and understand the contents of the above-identified specification, including the claims, as amended by any amendment referred to above.

We (I) acknowledge the duty to disclose information known to be material to the patentability of this application as defined in Section 1.56 of Title 37 Code of Federal Regulations.

We (I) hereby claim foreign priority benefits under 35 U.S.C. §119(a)-(d) or §365(b) of any foreign application(s) for patent or inventor's certificate, or §365(a) of any PCT International application which designated at least one country other than the United States, listed below and have also identified below, by checking the box, any foreign application for patent or inventor's certificate, or PCT International application having a filing date before that of the application on which priority is claimed. Prior Foreign Application(s)

Application No.	Country	Day/Month/Year	Priority Claimed
99202757.3	EP	26 AUGUST 1999	■ Yes ☐ No
			☐ Yes ☐ No
			☐ Yes ☐ No
			☐ Yes ☐ No

We (I) hereby claim the benefit under Title 35, United States Code, §119(e) of any United States provisional application(s) listed below.

(Application Number)	(Filing Date)
(Application Number)	(Filing Date)

We (I) hereby claim the benefit under 35 U.S.C. §120 of any United States application(s), or §365(c) of any PCT International application designating the United States, listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in the prior United States or PCT International application in the manner provided by the first paragraph of 35 U.S.C. §112, I acknowledge the duty to disclose information which is material to patentability as defined in 37 CFR §1.56 which became available between the filing date of the prior application and the national or PCT International filing date of this application.

Application Serial No.	Filing Date	Status (pending, patented, abandoned)

And we (I) hereby appoint Steven B. Kelber, Reg. No. 30,073; Jerold I. Schneider, Reg. No. 24,765; Paul C. Kimball, Reg. No. 34,641; Wilburn L. Chesser, Reg. No. 41,668; James M. Heintz, Reg. No. 41,828; Amy L. Miller, Reg. No. 43,804; Lisa K. Norton, Reg. No. 44,977; and Christopher W. Raimund, Reg. No. 47,258, as our (my) attorneys, with full powers of substitution and revocation, to prosecute this application and to transact all business in the Patent Office connected therewith; and we (I) hereby request that all correspondence regarding this application be sent to Supervisor, Patent Prosecution Services, Piper Marbury Rudnick & Wolfe LLP, 1200 Nineteenth Street, N.W., Washington, D.C. 20036-2412.

We (I) declare that all statements made herein of our (my) own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

Etienne DEGAND

NAME OF SOLE INVENTOR

E. Degand

Signature of Inventor

Le 28 février 2002

Date

Residence: Jumet, BELGIUM

BEL

Citizen of: BELGIUM

Post Office Address: c/o GLAVERBEL - Centre R. & D., Rue de l'Aurore, 2, B-6040 Jumet, BELGIUM